

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the application of:

Nelson, David., et al.

Serial No.: 10/822,573

Filing Date: April 12, 2004

For: EFFICIENT WATER FILTERS

Examiner: Ivars C. Cintins

Group Art Unit: 1724

Docket No. 482.152 US

DECLARATION OF EDWARD B. RINKER**UNDER 37 C.F.R. §1.132**

I, Edward B. Rinker, being duly sworn, hereby declare as follows:

1. I received a Bachelor of Science degree in Chemical Engineering from the University of California at Davis in 1992. I also received a Doctor's degree in Chemical Engineering from the University of California at Santa Barbara in 1997.
2. I am employed by The Clorox Company, owner by assignment of the above-identified patent application. I am one of the co-inventors in the above-identified patent application. I have worked in materials science research and development at Clorox since 1997, specializing in composite powders, plastics and fibers.
3. I have been working in the field of materials science and engineering since 1997.
4. I am the author of 15 publications in the field of chemical engineering.
5. I am familiar with the outstanding office action in the above-identified patent application. Claims 1-35 are pending and are rejected under 35 USC 103(a) in view of Kuennen et al. (US Patent No. 6,368,504, hereinafter '504). I am aware that the Examiner considers the present invention obvious in view of Kuennen and one of ordinary skill in the art at the time the invention was made.
6. '504 provides the following in column 1, line 60 to column 2, line 11:

"To increase filter performance at the expense of production yields and flow rates, another known carbon block manufacturer reduces the mean particle diameter of the carbon particles used to produce the block. To manufacture this carbon mixture, the carbon normally ground to form typical 80x325 mesh is subjected to a special grinding process that increases the level of carbon particles

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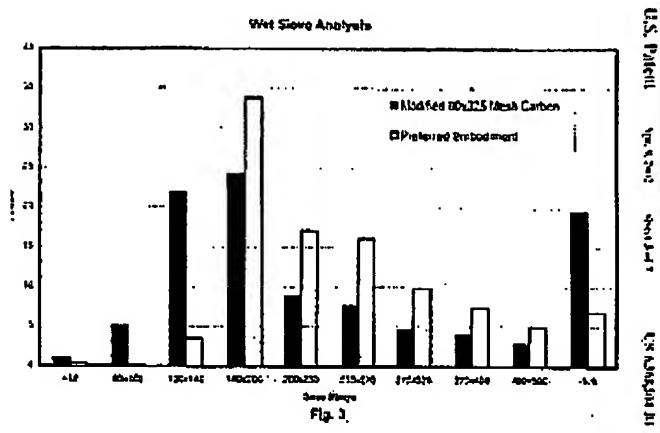
smaller than 325 mesh. Although the grinding operation inherently results in some variation, this modified carbon mixture generally provides a mean particle size of approximately 75 microns and a particle size distribution with approximately 25% or more of the carbon particles being larger than 140 mesh and 25% or more of the carbon particles being smaller than 500 mesh. Although the resulting carbon block filters provide improved performance, the high level of small carbon particles provides reduced flow rates and results in production losses of up to 20-30%. Further, the high level of small carbon particles produces carbon blocks that are relatively soft, making them susceptible to damage."

7. It is my opinion that reference to "a special grinding process" (col. 1, line 65) and "the grinding operation" (col. 1, line 67) in the '504 patent refer to the process by which the particles of the carbon block water filter are prepared by the "manufacturer". It is also my opinion that the grinding process described and used by the "manufacturer" is a single grinding process well known in the art. It is also my opinion that a single grinding process will result in a unimodal grain size distribution. It is furthermore my opinion that the '504 patent disclosure does not enable one skilled in the art to prepare a bimodal particle size distribution as claimed in the present application. The following aspects support the opinions made herein.
8. Particle size reduction is typically accomplished by a single grinder or a sequential series of grinders, which is well known in the art. All grinding operations yield unimodal distributions as long as the feed material is composed on a single type of material (e.g. activated carbon). The particle size reductions may vary over quite a wide range. It is not unusual for the particles of a suspension produced in a grinding operation, for example, to vary by a factor of 100 from the smallest to the largest size. Because the impact and shattering phenomena in a grinder are randomly distributed, the resulting particle size distribution is randomly distributed. A random distribution is represented by a Gaussian distribution. A Gaussian type distribution is characterized by a typical bell shaped curve.
9. It is well known in the art to describe particle size distributions with a graphical representation in the form of a histogram. The mode of a histogram is the largest class interval where maximum distribution occurs. The calculation of the mode for a

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distribution is straightforward, simply examine each class and select the one with the greatest frequency. However, histograms that truncate the largest and smallest particle size classes may show abnormally large bars. This is because they represent all classes of particles larger or smaller than the limits in the histogram. These bars are not modes.

10. The following is a representative histogram of a unimodal distribution, copied from Figure 3 of the '504 patent. The mean particle diameter of 75 micron (ie. 140x200 mesh) is shown with the highest peak, which represents the mode of the graph. This is a typical Gaussian distribution based on a single grinding process, which produces one mode.



(Copied from Fig. 3 of 6,368,504B1)

11. It is well known in the art that a bimodal distribution typically results from two different generation processes. A bimodal distribution might result from a process involving breakup of large particles with variable composition within the particles such as rocks containing minerals, multiple sources of particles or variable growth mechanisms in a system. Often, bimodality of the distribution may indicate that the sample is not homogenous and the observations come in fact from two or more "overlapping" distributions.

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12. The following is a representative graph of a bimodal distribution, copied from Figure 2A of the present application. Two modes are shown in the distribution; peak 220 and peak 230.

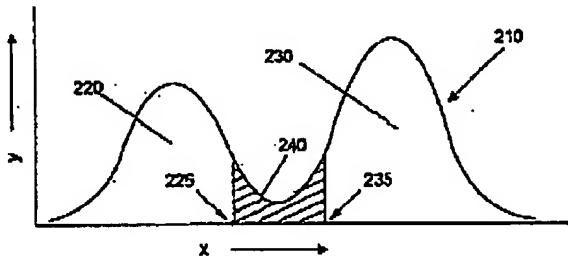


FIG. 2A
(Copied from Figure 2A of the present application)

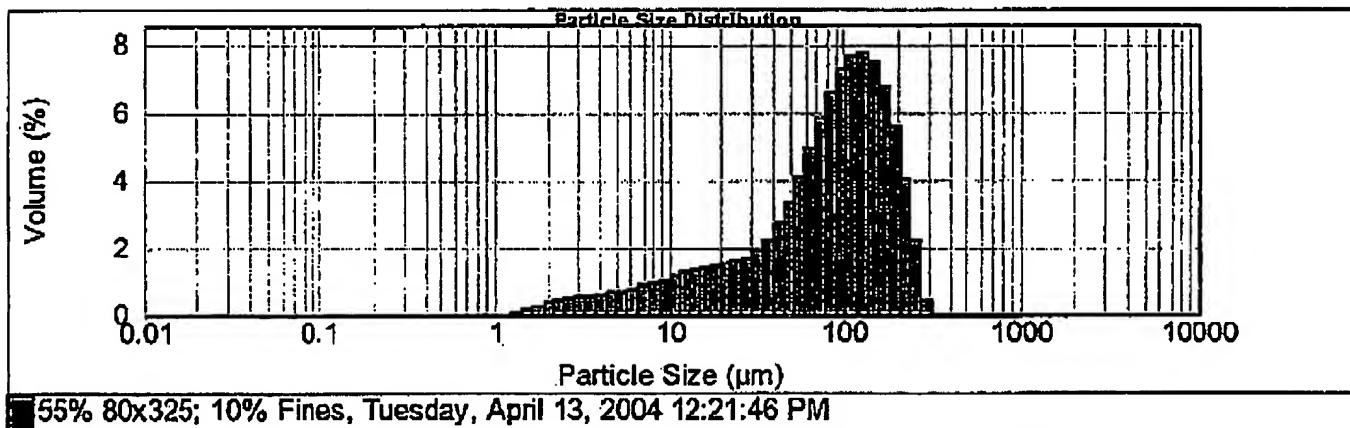
13. One method of developing a histogram is wet sieving. Wet sieving is very common, but yields crude results. This method involves collecting particles of various size ranges on sieves with different size ranges stacked on top of each other. Particles having sizes larger than the sieve aperture size are retained on the sieve and particles having sizes smaller fall through to the next sieve below and so on for any number of sieves. The smallest particles that pass through the sieve with the smallest aperture size are collected in a pan at the bottom of the sieve stack (ie. -500 mesh). The particles from each sieve and pan are then weighed and plotted on a histogram. For wide particle size distributions, for example a distribution with a large portion of particles larger than 80 mesh and smaller than 500 mesh, it is common that there will be higher weights of particles in the 80 mesh sieve and the -500 mesh pan. For this reason, it is difficult to represent the actual particle size distribution using the sieve method. As best understood, this observable fact is illustrated in Figure 3 of '504 presented above, wherein the peak at -500 is abnormally large because it is a compilation of all particles smaller than -500. Accordingly, the peak at -500 is not a mode. This is also apparent in Figures 3 and 7 of '504.

14. A more accurate method for measuring and depicting the particle size distribution of a powder is to use light scattering instruments such as the Malvern Mastersizer 2000. This method is used in the present application to graphically represent particle size

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distribution. This method is capable of measuring particle sizes in much smaller increments than sieves and can easily measure particles down to 1 micron in diameter (i.e. >5000 mesh). When particle size distributions are measured with light scattering techniques, it is easier for one to see the Gaussian distribution of particle sizes. The results are often plotted with a log scale on the x-axis (microns) and a linear scale on the y-axis (Volume %). Such plots are referred to as log-normal plots.

15. Below is an example of a log-normal plot of a typical 80x325 mesh carbon, prepared by using a single grinding operation. This is similar to the carbon samples discussed in '504. (Note: See Attachment 1 for further particle size parameters.)



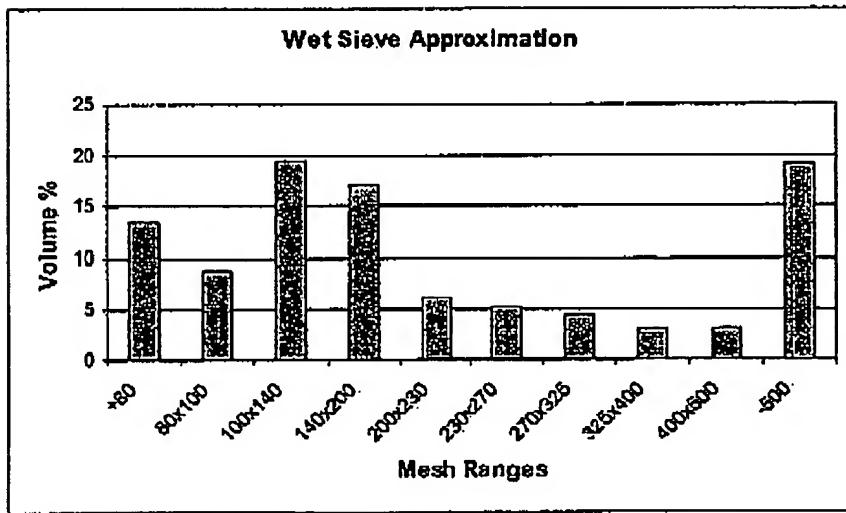
Histogram 1: Particle Size Distribution Measured by Malvern Mastersizer 2000

Histogram 1 shown above has a different shape compared to the histogram in Fig. 3 of '504, because the histogram above takes into account theoretical class sizes smaller than -500 mesh. The long tail represents a true theoretical picture of the high peak at -500 mesh depicted in Fig. 3 of '504. In order to make a direct comparison between the histogram above and the histogram of '504, one must determine the total volume percentage of particles in the same size classes as shown in Fig. 3 of '504. For example, the -500 size class can be determined by calculating the area under the curve between 1 micron (5000 mesh) and 30 microns (500 mesh). Similarly, the area under the curve between 30 microns (500 mesh) and 37 microns (400 mesh) will yield the volume

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percentage of particles between 400 and 500 mesh and so on. Using this technique, one can convert the log-normal plot of the particle size distribution shown above into a wet sieve histogram comparable to Fig. 3 of '504.

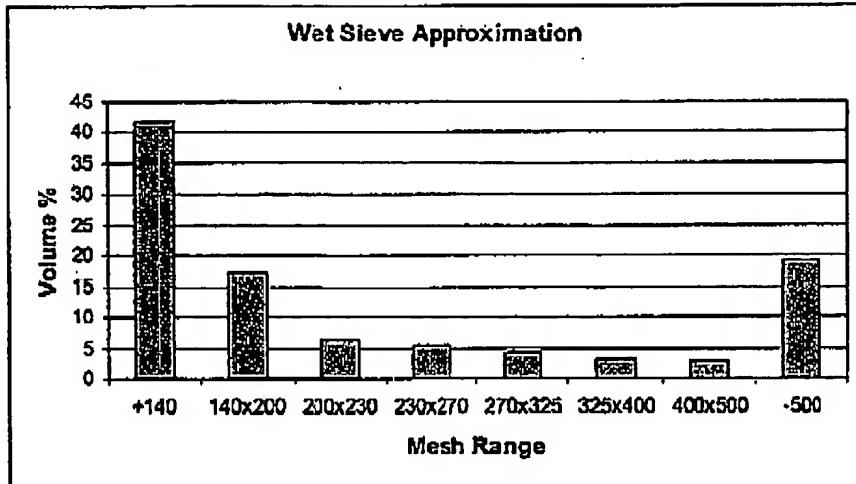
16. Provided below is a wet sieve histogram (Histogram 2) prepared by converting the log-normal plot of the typical 80x325 mesh particle size distribution shown above into a Histogram 1. Histogram 2 is comparable to Fig. 3 of '504. The distribution does not appear to be Gaussian or bell shaped, but it is. The bars on the end limits (ie. +80 or -500) are abnormally high because of the inclusion of all particles larger or smaller than the limit of mesh size identified in Fig. 3 of '504. The -500 peak contains all particle sizes between 5000 mesh and 500 mesh. Compared to the log-normal plot above, this is represented by the long leading tail (ie. From about 1 micron to about 30 microns) of the bell shaped curve.



Histogram 2

17. Provided below is Histogram 3 which shows the bars at +80, 80x100 and 100x140 of Histogram 2 grouped together into one bar. This directly represents the upper and lower size classes based on a typical 80x325 particle size distribution of a typical carbon sample.

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Histogram 3

18. Accordingly, Histogram 3 shows a typical carbon sample with more than 40% of the particles in the distribution having a particle size greater than 140 mesh and about 19% of the particles having a particle size less than 500 mesh. This illustratively shows a particle size distribution with approximately 25% or more of the carbon particles being larger than 140 mesh and 19% or more of the carbon particles being smaller than 500 mesh (see Col. 2, lines 4-6 of '504). The distribution is monomodal, because the high peaks at +140 and -500 are not true peaks, as discussed in detail above.

19. From my experience in the development of filtration media, I am intimately familiar with the affect of particle size distribution on filtration performance. It is common to use carbon with a unimodal distribution to manufacture carbon block filters. It is also common to use carbon with a large portion of particles smaller than 500 mesh (see Histogram 1 above with long leading tail). Carbon blocks made with carbon having a particle size distribution with a long leading tail tend to have lower water flow rates at a given pressure and also tend to clog prematurely. In the present invention, it was found that by mixing two carbonaceous materials with modes separated by more than 10 microns and having between 1 and 15 vol% of particles between the modes, that the resulting carbon blocks have improved flow rates and do not clog as easily as standard carbon blocks manufactured with unimodally distributed carbon (i.e. 80x325 mesh). This is particularly true when making very small carbon blocks, as described in the examples

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1-3 of the present application. Without being held to theory, it is believed that the bimodal distributions of the present invention leads to optimal packing density of the particles such that there are optimal interstitial spaces between the particles for adequate flow rates of water while also having adequate surface area of the carbon (related to particle size) to provide adequate contaminant removal. It is also believed that the two grain size modes interfere with the package geometry of the particles, leaving the structure more open for flow of water but not too open that the filter no longer removes contaminants to the proper levels. A unimodal particle size distribution gives a more uniform packing density and therefore results in a tighter carbon block filter with inadequate flow rate.

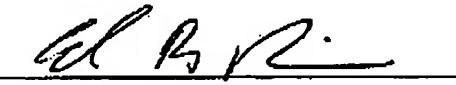
20. The '504 patent provides the following "a mean particle size of approximately 75 microns and a particle size distribution with approximately 25% or more of the carbon particles being larger than 140 mesh and 25% or more of the carbon particles being smaller than 500 mesh". '504 also provides that "[t]o manufacture this carbon mixture, the carbon normally ground to form typical 80x325 mesh is subjected to a special grinding process that increases the level of carbon particles smaller than 325 mesh." It is my opinion that based on this disclosure, one skilled in the art would use a single grinding process and a unimodal particle distribution would be the result. In fact, even if the block of the prior art was manufactured used a series of single grinding processes, a unimodal distribution would still be the result. Thus, it is my opinion that the only way to make a bimodal distribution is to have two separate grinding operations with two separate feed materials and mixing the resulting ground materials.
21. Histogram 3 presented above is a graphical representation of how the prior art described could be plotted. The distribution is unimodal. The '504 patent disclosure does not teach, disclose or suggest a multimodal system.
22. In the present application, two separate feed materials were mixed together to prepare the resulting bimodal particle size distribution. Examples 1, 2 and 3 illustrate the method used. Accordingly, Example 3 provides that 32 vol % activated carbon (80 x 325 mesh) and 24% activated carbon (-325 mesh) were mixed together to make an embodiment of the present invention.

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23. In conclusion, the '504 disclosure does not address the grinding process used by the prior art in detail. '504 makes reference to "a special grinding process" in col. 1, lines 65-66. Figures 2, 3 and 7 show histograms depicting unimodal distributions of the preferred embodiments of Kuennen and the prior art. A single grinding process would produce a unimodal distribution. There is no teaching or suggestion in '504, specifically in col. 1, line 60 to col. 2, line 11, or Figures 2, 3 and 7, as to how to prepare filtration media with a grain size distribution that is bimodal. The reference in '504 to the particle size distribution "approximately 25% or more of the carbon particles being larger than 140 mesh and 25% or more the carbon particles being smaller than 500 mesh" does not anticipate or render obvious the particle size distribution claimed in the present application, namely "[a] water treatment device, comprising filtration media containing a volume of grains having a multiply modal grain size distribution that has at least a first mode and a second mode, wherein the first mode includes a first grain size and the second mode includes a second grain size; wherein the grains of the first mode are smaller than the first grain size and the grains of the second mode are larger than the second grain size; wherein between about 1 and 15 vol % of the grains have a grain size in a range between the first grain size and the second grain size."

24. I, EDWARD B. RINKER, declare that the foregoing statements of fact are true and correct of my knowledge; that statements made on information and belief are believed to be true; and that willful false statements and the like so made are punishable by fine or imprisonment or both under §1001 of Title 18, U.S. Code, and may jeopardize the validity of this application or any patent issuing thereon.

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EDWARD B. RINKER

Attachment 1

Ed Rinker Declaration

Particle Size Parameters for Histogram 1

Size (µm)	Curr In %	Dif %	Size (µm)	Curr In %	Dif %	Size (µm)	Curr In %	Dif %	Size (µm)	Curr In %	Dif %	Size (µm)	Curr In %	Dif %
0.010	0.00	0.00	0.162	0.00	0.00	3.311	0.58	0.00	60.258	4.95	0.00	1096.478	0.00	0.00
0.011	0.00	0.00	0.208	0.00	0.00	3.802	0.60	0.00	69.183	5.80	0.00	1258.825	0.00	0.00
0.013	0.00	0.00	0.240	0.00	0.00	4.365	0.64	0.00	78.433	6.80	0.00	1445.440	0.00	0.00
0.015	0.00	0.00	0.275	0.00	0.00	5.012	0.69	0.00	91.201	7.28	0.00	1659.567	0.00	0.00
0.017	0.00	0.00	0.318	0.00	0.00	5.754	0.75	0.00	104.713	7.69	0.00	1905.461	0.00	0.00
0.020	0.00	0.00	0.363	0.00	0.00	6.607	0.83	0.00	120.226	7.80	0.00	2187.762	0.00	0.00
0.023	0.00	0.00	0.417	0.00	0.00	7.588	0.92	0.00	138.038	7.51	0.00	2511.888	0.00	0.00
0.025	0.00	0.00	0.479	0.00	0.00	8.710	1.02	0.00	158.489	6.78	0.00	2884.032	0.00	0.00
0.030	0.00	0.00	0.550	0.00	0.00	10.000	1.12	0.00	181.970	5.57	0.00	3311.311	0.00	0.00
0.035	0.00	0.00	0.631	0.00	0.00	11.482	1.22	0.00	208.930	4.01	0.00	3801.694	0.00	0.00
0.040	0.00	0.00	0.724	0.00	0.00	13.183	1.31	0.00	239.683	2.22	0.00	4365.158	0.00	0.00
0.048	0.00	0.00	0.832	0.00	0.00	15.136	1.58	0.00	275.423	0.39	0.00	5011.872	0.00	0.00
0.052	0.00	0.00	0.955	0.00	0.00	17.378	1.44	0.00	318.228	0.00	0.00	5754.399	0.00	0.00
0.060	0.00	0.00	1.098	0.00	0.00	19.953	1.48	0.00	363.078	0.00	0.00	6506.934	0.00	0.00
0.069	0.00	0.00	1.259	0.02	0.00	22.809	1.54	0.00	416.889	0.00	0.00	7585.778	0.00	0.00
0.079	0.00	0.00	1.445	0.13	0.00	25.303	1.66	0.00	478.030	0.00	0.00	8709.636	0.00	0.00
0.091	0.00	0.00	1.680	0.24	0.00	30.200	1.85	0.00	549.541	0.00	0.00	10000.000		
0.105	0.00	0.00	1.905	0.32	0.00	34.674	2.19	0.00	630.957	0.00	0.00			
0.120	0.00	0.00	2.168	0.40	0.00	38.811	2.68	0.00	724.436	0.00	0.00			
0.135	0.00	0.00	2.512	0.48	0.00	45.709	3.34	0.00	831.784	0.00	0.00			
0.158	0.00	0.00	2.884	0.51	0.00	52.481	4.11	0.00	954.993	0.00	0.00			
0.182	0.00	0.00	3.311			60.256			1096.476					

Mesh No	Aperture µm	Volume In %	Vol Below %	Mesh No	Aperture µm	Volume In %	Vol Below %	Mesh No	Aperture µm	Volume In %	Vol Below %
10	2000	0.00	100.00	35	500	0.00	100.00	120	125	9.74	67.91
12	1680	0.00	100.00	40	420	0.00	100.00	140	105	9.18	59.17
14	1410	0.00	100.00	45	354	0.00	100.00	170	88	7.90	48.99
16	1190	0.00	100.00	50	297	1.73	98.98	200	74	6.17	41.08
18	1000	0.00	100.00	60	250	4.89	98.25	230	63	5.34	34.91
20	841	0.00	100.00	70	210	7.03	93.56	270	53	4.40	29.57
25	707	0.00	100.00	80	177	8.80	86.53	325	44	3.10	25.17
30	695	0.00	100.00	100	148	9.82	77.73	400	37		22.07
35	500	0.00	100.00	120	125	67.91					